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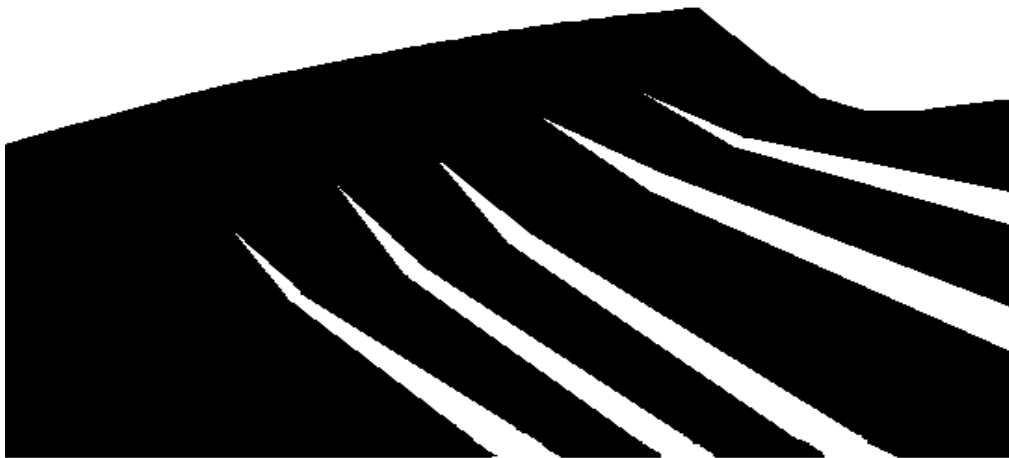
January 9, 1997

LANL-CST-DP-109, R0

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## PARTICLE SIZE REDUCTION OF SAMPLES

### ***LOS ALAMOS QUALITY PROGRAM***



#### APPROVAL FOR RELEASE

J. T. FABRYKA-MARTIN - PREPARER

Signature on file

DATE

Date on file

J. T. FABRYKA-MARTIN - PRINCIPAL INVESTIGATOR

Signature on file

DATE

Date on file

M. J. CLEVINGER - QUALITY ASSURANCE PROJECT LEADER

Signature on file

DATE

Date on file

**Los Alamos**

Yucca Mountain Site  
Characterization Project

## HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	01/09/97	N/A	Initial procedure.

**Los Alamos**

Yucca Mountain Site  
Characterization Project

# **PARTICLE SIZE REDUCTION OF SAMPLES**

## **1.0 PURPOSE**

This detailed technical procedure (DP) describes the procedure for reducing the particle size of rock samples prior to leaching them for chemical and isotopic analyses for the Yucca Mountain Site Characterization Project (YMP).

## **2.0 SCOPE**

This DP applies to YMP personnel who process rock samples for chemical analyses as part of the YMP's Water Movement Test task for Los Alamos National Laboratory (LANL).

## **3.0 REFERENCES**

LANL-YMP-QP-02.7, Personnel Training  
LANL-YMP-QP-03.5, Documenting Scientific Investigations  
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards  
LANL-CST-DP-92, Sample Leaching to Extract Soluble Chloride and Bromide  
LANL-CST-DP-103, Identification, Storage, and Handling of Samples for the Water Movement Test

## **4.0 DEFINITIONS**

### **4.1 Rock Samples**

Rock samples are materials intended for laboratory studies or analyses that were obtained directly from the Yucca Mountain area or obtained from special sources such as prototype testing localities. Rock samples may include, but are not limited to, surface samples, drill cores, drill cuttings, and rocks from underground excavations.

## **5.0 RESPONSIBILITIES**

The following personnel are responsible for the activities identified in section 6.0 of this procedure:

- Principal Investigator (PI) for the Water Movement Test
- YMP personnel performing work to the procedure

## 6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified or replaced by a new document, or until current work practice is documented in accordance with QP-03.5, subsection 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

### 6.1 Principle

This DP describes methods for reducing the particle size of rock and mineral samples to increase the available surface area and efficiency of the leaching process such as that used in DP-92 to extract halides and other soluble constituents from samples. Subsection 6.5.2 describes the procedure for breaking rock samples into smaller pieces. Subsection 6.5.3 describes the procedure for separating secondary minerals from bulk matrix rocks and then crushing and pulverizing these mineral separates prior to additional processing.

### 6.2 Equipment and Hardware/Software

Equipment which may be useful for reducing the particle size of samples is listed below. Not all of the items are required. Items equivalent to those listed may be used provided they perform the same function with an acceptable level of performance as judged by the user.

Supplies for cleaning equipment (subsection 6.5.1):

- water purification system capable of producing deionized (DI) water with resistivity greater than 17.5 megohms-cm (calibration not required; see section 6.3.2)
- 8M nitric acid

Equipment used to reduce particle size of rocks (subsection 6.5.2):

- steel plate to provide surface for crushing rocks (e.g., 12-in diameter by 1-in thick)

- hammer with heavy head (e.g., 2-lb)
- sturdy box large enough to contain steel plate (e.g., about 24 x 18 x 12-in)
- disposal clean-room gloves (at least 2 pairs per sample),
- one sturdy plastic bag per sample, for lining inside of box (e.g., 30-gallon, 6 to 8 mil in thickness)

Equipment used to separate and crush secondary minerals (subsection 6.5.3):

- motorized ball grinder with ball and bowl made out of diatomite
- short-wave ultraviolet (UV) light

#### 6.2.1 Equipment Malfunction

Any equipment malfunction occurring during implementation of this procedure is likely to be readily detectable in the course of conducting work and hence is not expected to have a detrimental effect on the final results. The water purification system has a meter that indicates the resistivity of the purified water. This meter is checked before each use to verify that the resistivity is within the range of acceptable values, i.e., greater than 17.5 megohm-cm. If a problem with any equipment arises which can be considered a potential source of error or uncertainty for the results, then it is addressed following section 6.7.

#### 6.2.2 Safety Considerations

Good laboratory and scientific practices are used in the laboratory to protect against injury, such as eye protection when working with a hammer or acid. Applicable LANL and/or LANL-contractor safety practices for conducting laboratory work are followed.

#### 6.2.3 Special Handling

Care must be taken to minimize any potential for contamination of the sample with the analyte of interest (usually chloride or  $^{36}\text{Cl}$ ).

### 6.3 Preparatory Verification

#### 6.3.1 Hold Points

N/A

#### 6.3.2 Calibration

The water purification system does not require calibration because the purpose of its meter is limited to alerting the user to a failure of the

system to produce deionized water, as indicated by the reading on the resistivity meter. Meter failure is indicated by a “zero” reading, at which time the meter is replaced by the vendor.

#### 6.3.3 Environmental Conditions

Measures to minimize the potential for cross-contamination by particles or sources of chloride or other analytes of interest other than those in the sample being processed are discussed in section 6.7. Work is conducted in a room with adequate ventilation. No other special environmental conditions are required.

### 6.4 Control of Samples

Sample identification and control must be sufficient to trace a sample and its derivatives from its original field location to the point of analysis, and the integrity of the sample must be safeguarded during the entire process. Consequently, users must be trained to DP-103 before they can work with YMP samples and must follow guidelines set forth in that document for sample control.

### 6.5 Implementing Procedure

#### 6.5.1 Cleaning

Conduct the following steps before using equipment for the first time for processing samples:

6.5.1.1 Wearing a pair of disposable, clean-room gloves, use 8M nitric acid to rinse the parts of any equipment that will be in contact with the sample.

6.5.1.2 After the acid rinse, rinse the parts with DI water. Then soak all parts in DI water for 12 hours, and air dry.

#### 6.5.2 Size Reduction of Rock Samples

6.5.2.1 Put on two pairs of disposable clean-room gloves. If not already done, clean hammer and steel plate with DI water, followed by sponging the surface and rinsing it with DI water. Let air dry.

6.5.2.2 Line box with a plastic bag, split down one side.

6.5.2.3 Put precleaned steel plate on top of liner, in bottom of box.

6.5.2.4 Separate out large pieces (> ~2 cm dimension) for crushing, and put on top of steel plate. Do not create more than one layer of smaller pieces, or work on more than one large chunk at a time.

- 6.5.2.5 Use precleaned hammer to manually pulverize rock on top of plate.

**NOTE:** The crushed material should consist of pieces with approximate dimensions of 0.25 to 2-cm for dense rock, and somewhat less (maximum dimensions of about 0.5 cm) for poorly consolidated material such as fault gouge or breccia. The user should try to minimize the proportion of material that is less than about 0.25 cm. These size dimensions are only intended as crude guidelines and do not require quantitative verification.

- 6.5.2.6 Lift out steel plate and transfer crushed sample into a container prelabelled with the sample ID.

- 6.5.2.7 Repeat steps 6.5.2.4 to 6.5.2.6 until sample is completely pulverized, or until sufficient coarse material for analysis has been accumulated (about 1-5 kg).

- 6.5.2.8 Clean hammer and steel plate with DI water, followed by sponging the surface, then rinsing with DI water. Let air dry. In order to keep the equipment clean, either store it dry in a clean lab with class 100 air, or else store it in a plastic bag, leaving the bag open to prevent rusting of the equipment.

- 6.5.2.9 Repeat steps 6.5.2.1 to 6.5.2.8 for each subsequent sample.

### 6.5.3 Separation, Crushing and Pulverization of Mineral Separates

- 6.5.3.1 For samples in which the minerals to be separated are calcite and opal, the user can distinguish these minerals by their fluorescence under short-wave UV light: calcite is nonfluorescent or fluoresces weakly to strongly, usually blue-white, while many opals fluoresce yellow-green. The user can separate a sample by this technique into opal-rich, calcite-rich, or mixed opal-calcite portions that may also correspond to different parts of a paragenetic mineral sequence (e.g., an early deposit and a later deposit sitting on it). Alternatively, the user may take a bulk representative sample which will generally consist of a mixture of opal and calcite. In any case, the user documents the approach used to separate mineral phases.

- 6.5.3.2 Preleach bulk sample pieces in DI water for 12 hours and discard leachate.

- 6.5.3.3 Using a scrub brush, wash off residual drilling mud and debris, and rinse sample with DI water.

- 6.5.3.4 Sonicate sample in a beaker of DI water for 10 minutes, making sure the water covers the minerals.

- 6.5.3.5 Remove from ultrasonic bath, discard water and rinse again with DI water.
- 6.5.3.6 Air-dry sample in an open plastic bag.
- 6.5.3.7 If necessary, follow sample breakup procedure described in subsection 6.5.2 to reduce the sample to an appropriate size (<0.5 cm in dimension) to be placed in the grinding device.
- 6.5.3.8 Precontaminate grinding device by pulverizing 5-10 grams of crushed sample in it for 5 minutes. Discard this sample.
- 6.5.3.9 Pulverize the sample, 100-200 grams at a time for 5 minutes each, emptying the device after each pulverization step into a container prelabelled with the sample ID. The pulverized sample should have the consistency of powder. Continue pulverizing additional material until a sufficient quantity has been obtained for processing such as leaching for soluble analytes following DP-92.
- 6.5.3.10 Clean the parts of the grinding devices that were in contact with the sample with 8M nitric acid. After the acid rinse, rinse all parts with DI water, and air dry.
- 6.5.3.11 Repeat steps 6.5.3.1 through 6.5.3.10 for each new sample.

## 6.6 Data Acquisition and Reduction

N/A

## 6.7 Potential Sources of Error and Uncertainty

If adequate precautions are not taken, then Cl,  $^{36}\text{Cl}$  or other constituent could inadvertently be introduced into the sample as a contaminant. Hence, the user wears gloves during sample processing, and minimizes cross-contamination between samples by cleaning devices between samples. Careful labeling of containers reduces errors due to mislabeling. The user is responsible for documenting any problems which could be considered potential sources of error or uncertainty for the results, in accordance with QP-03.5.

## 7.0 RECORDS

Records to be generated as a result of the proper execution of this DP are entries in laboratory notebooks and in the Sample Inventory Logbook. These records are controlled by procedures QP-03.5 and DP-103.

## 8.0 ACCEPTANCE CRITERIA

The criteria that show that this procedure has been correctly implemented are the records identified in Section 7.0.

## **9.0 TRAINING**

A prerequisite for this DP is to train to DP-103. This DP requires read-only training. Training of personnel to this DP is documented pursuant to QP-02.7.

## **10.0 ATTACHMENTS**

N/A